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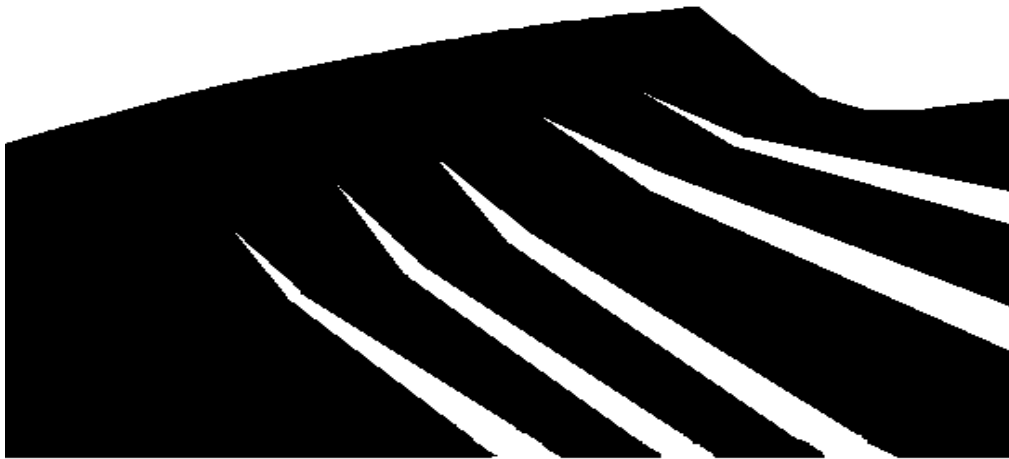
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MEASUREMENT OF MOISTURE CONTENT OF SOIL SAMPLES

LOS ALAMOS QUALITY PROGRAM



APPROVAL FOR RELEASE

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Los Alamos

Yucca Mountain Site

Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	01/06/92	N/A	Not Applicable.
R1	06/15/92	5-8	Major revisions address labeling of containers and criteria for monitoring moisture loss from samples.
R2	01/09/97	All	Revised to comply with LANL-YMP-QP-06.3 requirements.

Los AlamosYucca Mountain Site
Characterization Project

MEASUREMENT OF MOISTURE CONTENT OF SOIL SAMPLES

1.0 PURPOSE

This detailed technical procedure (DP) describes the process for determining moisture content of soil samples for the Yucca Mountain Site Characterization Project (YMP).

2.0 SCOPE

This DP applies to all YMP personnel who conduct determinations of moisture content of soil samples as part of the YMP's Water Movement Test task for Los Alamos National Laboratory (LANL).

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-CST-DP-89, Procedure for Sieving Soil and Rock Samples
LANL-CST-DP-92, Sample Leaching to Extract Soluble Chloride and Bromide
LANL-CST-DP-103, Identification, Storage, and Handling of Samples for the Water Movement Test
LANL-CST-DP-108, Collection of Field Samples for the Water Movement Test

4.0 DEFINITIONS

4.1 Soil

Soil includes any unconsolidated material above bedrock, i.e., the regolith. Generally, the samples processed by this DP will have been collected by the methods described in DP-108 for the express purpose of moisture determinations.

4.2 Gravimetric Moisture Content

Gravimetric moisture content refers to the mass of water evolved from a soil sample during heating following the procedure described in this DP.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principal Investigator (PI) for the Water Movement Test
- YMP personnel performing work to the procedure

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified or replaced by a new document, or until current work practice is documented in accordance with QP-03.5, Section 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

6.1 Principle

Moisture contents of soil samples may need to be determined in order to use the chloride or chlorine-36 results to estimate infiltration rates, as part of the YMP Water Movement Test. A standardized procedure for moisture content determination ensures the consistency and integrity of these analytical results. The accuracy of water content determinations depends upon reproducible conditions of collection, weighing, and drying.

6.2 Equipment and Hardware/Software

Equipment needed to conduct moisture content determinations is listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the user or the PI.

- analytical balance, with minimum of 500 gram capacity and 0.01 gram sensitivity
- convection oven capable of maintaining the temperature at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$

Materials and supplies used in this procedure include airtight moisture containers designed to hold samples for soil moisture analysis, electrical tape or parafilm to provide a moisture-proof seal, indelible markers to label the moisture containers, tongs or potholders to remove hot samples from the oven, and a commercial-quality thermometer with $\pm 1^{\circ}\text{C}$ precision for monitoring oven temperature.

6.2.1 Equipment Malfunctions

Any equipment malfunction occurring during implementation of this procedure is likely to be readily detectable in the course of conducting work and hence is not expected to have a detrimental effect on the final results. If a problem with any equipment arises which can be considered a potential source of error or uncertainty for the results, then it is addressed following section 6.7.

6.2.2 Safety Considerations

Good laboratory and scientific practices are used to protect against operator injury. To prevent burns, tongs, potholders, or the equivalent are recommended when removing samples from the oven. Applicable LANL and/or LANL-subcontractor safety practices for conducting laboratory work are followed, as appropriate.

6.2.3 Special Handling

Care must be taken to minimize any potential for moisture gain or loss from the sample before its initial (wet) weight has been obtained. Precautionary measures are described in DP-108 and section 6.5.3.2.

6.3 Preparatory Verification

6.3.1 Hold Points

N/A

6.3.2 Calibration

The balance used to weigh empty sample containers and to weigh soil samples before and after drying is controlled pursuant to QP-12.3.

6.3.3 Environmental Conditions

Determination of moisture content is performed in a laboratory.

6.4 Control of Samples

It is imperative that sample identification and control be sufficient to trace a sample and its derivatives from its original field location to the point of analysis and that the integrity of the sample be safeguarded during the entire analytical process. Consequently investigators must be trained to DP-103 before they can work with samples analytically, and they must also follow guidelines set forth in that document for sample control.

6.5 Implementing Procedure

6.5.1 Preweighed Sample Containers

Airtight moisture containers are used for moisture determinations. Prior to use, each container is weighed and labeled with a unique identifier using an indelible marker.

6.5.2 Sample Collection

Sample collection is done in accordance with DP-108. The user records the sample identifier for each labeled container if it is different from the container identifier.

6.5.3 Gravimetric Moisture Determination

6.5.3.1 Outside of Container

Any tape or parafilm is removed from the container. If necessary, the outside of the sample container is wiped dry of any moisture which may have condensed on it during storage in the cooler. The containers are checked to ensure that identifiers are clearly legible.

6.5.3.2 Initial (Wet) Weight

The sample and container are weighed to the nearest 0.01 g or better on a calibrated balance as soon as possible after collection. Once this weight is obtained, the sample no longer needs to remain sealed and/or stored in a cooler.

6.5.3.3 Oven-drying

The container is placed in the convection oven and heated at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for at least 24 hours. After removal from the oven, the container is cooled until it is no longer hot to the touch (usually, this takes about 30 minutes). The cooled sample is weighed, and the container is returned to the oven for at least 6

hours. The sample is again cooled and reweighed. The drying/cooling/weighing cycle is repeated until a constant sample weight (i.e., within 0.1 g of the previous weight) is obtained. The time and result of each weighing are recorded in the laboratory notebook.

If multiple samples are to be analyzed, it is advisable to process them as a batch and place them in the oven together. Once samples are in the oven drying, precaution is taken to avoid adding wet samples during the drying period. Also, additional drying time is necessary if the oven is heavily loaded with samples, particularly if they are very damp.

6.5.4 Disposition of the Sample

The PI is consulted for disposition of the sample after its moisture determination and may direct the user to discard the sample or to reserve it for chloride or bromide analysis, pursuant to DP-92. If the soil sample contains a significant quantity of gravel or stones, then the PI may request that the sample be sieved following DP-89. If the sample is sieved, then the results are reported on both a bulk basis as well as a gravel-free basis (see section 6.5.5).

6.5.5 Calculation of Gravimetric Moisture Content

The weight loss during heating is assumed to be due solely to loss of water. Percent moisture is reported on a mass-of-water per dry-mass-of-sample basis. The calculation is as follows:

$$\begin{aligned}\% \text{ moisture content} &= [M_{\text{water}} / M_{\text{dry}}] \times 100 \\ &= [(M_{\text{wet}} - M_{\text{dry}}) / M_{\text{dry}}] \times 100\end{aligned}$$

where: M_{water} = mass of water, i.e., weight loss after drying

M_{dry} = mass of dry sample

M_{wet} = initial (wet) mass of sample

If the sample is sieved following moisture determination, then results are also reported on a gravel-free basis. In this case, the calculation is:

$$\begin{aligned}\% \text{ moisture content} &= [M_{\text{water}} / M_{\text{dry}<2\text{mm}}] \times 100 \\ \text{on gravel-free basis} &= [(M_{\text{wet}} - M_{\text{dry}}) / M_{\text{dry}<2\text{mm}}] \times 100\end{aligned}$$

where: $M_{\text{dry}<2\text{mm}}$ = mass of dry sample passing 2 mm sieve in DP-89

Results of these calculations are recorded in the laboratory notebook.

6.6 Data Acquisition and Reduction

A checklist for laboratory notebook entries required for this DP is provided in Attachment 1. Weights are recorded to the nearest 0.01 g or better. The acceptability and precision of the data are evaluated by the PI, taking into account the precision of the instruments used as documented pursuant to QP-12.3.

6.7 Potential Sources of Error and Uncertainty

A source of uncertainty is the limited analytical precision of the balance to resolve the difference between the initial (wet) weight and oven-dry weight of small samples. Careful labeling of containers reduces the possibility of errors due to mislabeling. Loss of moisture prior to determination of the initial wet weight is a potentially significant source of error. To minimize this possibility, this procedure requires that the sample be kept in a closed, air-tight container prior to determination of moisture content and that the sample be weighed as soon as possible after field collection. If a problem occurs which could pose a potential source of error or uncertainty for the results, then the user documents it in the laboratory notebook in accordance with QP-03.5.

7.0 RECORDS

Records to be generated as a result of the proper execution of this DP are entries in laboratory notebooks and in the Sample Inventory Logbook. These records are controlled by procedures QP-03.5 and DP-103.

8.0 ACCEPTANCE CRITERIA

The criteria that show that this procedure has been correctly implemented are the records identified in Section 7.0.

9.0 TRAINING

A prerequisite for this DP is to train to DP-103. This DP requires read-only training. Training of personnel to this DP is documented pursuant to QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Checklist of Laboratory Notebook Entries (1 page)

CHECKLIST OF LABORATORY NOTEBOOK ENTRIES

Initial descriptive information is entered in the laboratory notebook as appropriate prior to starting a technical procedure and on a continuing basis as experimental and procedural changes dictate. These entries are:

- reference to this DP, including revision number
- unique identifiers for any calibrated equipment used to conduct this DP

Additional information is entered into the laboratory notebook a minimum of once a day for each day that relevant laboratory work is done, and more often if conditions change. These entries are:

- date of entry
- list of samples processed, including their unique identifiers assigned according to DP-103
- problems (if any) which could be considered potential sources of error or uncertainty for the results

In addition to general entries, several parameters need to be recorded in order to evaluate the resulting data. Weights are recorded to the nearest 0.01 gram or better. These entries are:

- weight of empty container
- initial weight of sample before drying
- time at which sample is initially put into oven for drying
- time at which sample is removed from oven for weighing
- weight of cooled sample each time it is removed from oven
- weights of sieved fractions, if applicable
- results of calculations of moisture content